Stabilization of Industrial by-products using alkali activation

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Stabilization of Industrial by-products using alkali activation

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May 2015

National Institute of Technology, Rourkela

CERTIFICATE

This is to certify that the thesis entitled *"**Stabilization of Industrial by-products using alkali activation**"* submitted by ***Miss Jayashree Sahoo*** in partial fulfillment of the requirement for the award of ***Master of Technology (Dual Degree)*** in ***Civil Engineering*** with ***Geotechnical Engineering Specialization*** at National Institute of Technology Rourkela is an authentic work carried out by her under my guidance and supervision.

To the best of my knowledge, the matter presented in this thesis has not been presented in any other university/college for any other degree or diploma.

Date:

Place: ***Rourkela***

Prof. S P Singh

Department of Civil Engineering

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Jayashree Sahoo

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List of Abbreviations

Al= Aluminium

FA= Fly ash

GGBFS= Ground granulated blast furnace slag

NaOH= Sodium hydroxide

Si= Silicon

UCS= Unconfined strength

RHA= Rice husk ash

RM= Red Mud

XRD= X-ray Diffractometer

Abstract

With increase in the growth of industrialization, proper disposal of wastes is one of the major issues nowadays that has to be handled with seriously. An innovative solution that is efficient and environment friendly is required to tackle this problem of disposal. Research has sprung to full pace in recent years on how to utilize these waste materials in an effective way. Geopolymerization is the latest trend in that path. The basic requirements for the synthesis of any geopolymer are alkali solution and aluminosilicate material. So the by-products which are rich in Silica and Alumina can be used as aluminosilicate sources. In the present work, four by-products namely ground granulated blast furnace slag (GGBFS), fly ash, rice husk ash (RHA) and red mud were taken as raw materials. All the materials except rice husk ash constitute alumina along with silica. Rice husk ash, being rich in silica, was taken as one of the raw material to investigate the effects of NaOH on it. These four materials were activated by NaOH with varying percentages i.e. 5%, 10%, 15% of raw material. Light compaction tests were conducted for each addition of chemical to the raw material. The effect of different percentages of NaOH on the strength of raw materials was investigated by unconfined compressive tests. For each combination of raw material and percentage of NaOH, three samples were prepared at optimum moisture content (OMC) and maximum dry density (MDD) and kept for curing at ambient temperature for prefixed periods (i.e. 0day, 7days, and 28 days). Mineralogical analysis of the samples was done by X-ray diffraction (XRD) analysis. The results show that with increase in percentages of NaOH, the strength of GGBFS and fly ash increases. But the strength of alkali activated RHA on 7^{th} day of curing decreases from the immediate strength due to presence of carbon in the material. The strength of red mud with 5% NaOH on 7^{th} day is higher than the immediate strength and for other

percentages, the strength decreases. The reaction between alkali and slag is quick which completes with 7 days of curing. Reaction products were found to be sodium aluminium silicate in GGBFS, fly ash, and RHA from XRD analysis.

Keywords: Alkali Activation, Compaction test, UCS test, XRD analysis

Chapter 1
Introduction

With the tremendous growth of industrialization, the production of by-products (such as coal ash, red mud, slag etc.) from the industries also increases. Disposal of waste product is the biggest problem as it requires big expanse of land. Waste products may pollute this land by leaching toxic ingredients as well as pollute the air due to the presence of fine particles in the so called waste products. Today, researchers are attempting to utilize the waste products in construction works to tackle the environmental effects they cause. There is no proper way for 100% utilization of the waste products even today. Blast furnace slag and fly ash are popular resources for construction materials. GGBFS generally replaces cement in the making concrete and fly ash has been known as pozzolanic materials. Annual production of slag in India is around 21-24 million tons. In thermal power plants, coal is used as major source of energy. In India, more than 300 million-tons of coal are consumed by thermal power plants and produce closely 100,000 MW power. On the burning of coal, fly ash and bottom ash are generated as solid remains. Electrostatic precipitators collects fly ash in thermal plants. Around 163.56 million-tons of fly-ash is produced annually; out of which only 61.37% is being utilized for construction of roads and embankments, production of cement, mine-filling, reclamation of low-lying areas, agriculture, making bricks and tiles and others (CEA Annual Reports, 2012-13). The unused ash results in many environmental problems such as land pollution and deterioration of air and water quality. Also, the storage of fly ash demands considerable land for disposal with substantial chance of pollution of surrounding water/agricultural land due to overspill of by-products ash ponds during heavy precipitation. Red mud is a byproduct produced from the bauxite ore during alumina extraction in bayer process. Present estimated production of red mud is to be above three billion tons with a rough growth rate of 120 million tons per annum. The future management of red mud is of increasing environmental concern. Rice husk ash is an agricultural by-product produced during the burning of rice husk. Annual production of rice husk is about 108 tons in the world. Rice husk constitutes 20% of the 500 million tons of paddy produced

in the world. About 78 % of weight is received as rice, broken rice and bran during milling of paddy. Remaining 22 % of the weight of paddy is received as husk. This husk is used as fuel in the rice mills in the boiling process. Almost 75 % of husk contains volatile matter and rest 25 % of the weight of this husk is changed into ash in the course of the burning process, is known as rice husk ash (RHA). This RHA in turn comprises around 85 % - 90 % amorphous silica and remaining is carbon. India is a major rice producing country. Around twenty million tons of RHA are produced each year. This RHA is a big environmental problem affecting the land and the nearby area in which it is discarded. Various ways are being explored for the utilization of RHA by preparing it for commercial use. The properties of rice husk ash are influenced significantly by whether the husks had gone through complete destructive distillation or had only been incompletely burnt. RHA in extremely reactive form are opted as an appropriate raw material for making hydraulic cement. Many researchers are trying to explore the usability of these waste products by involving latest technologies. One of trending technologies is Geopolymerization. This word was coined by Joseph Davidovits in late 1970s. Geopolymer is an inorganic polymer with SiO_4 and AlO_4 tetrahedra being the structural units (Davidovits 1994). Geosynthesis is based on the ability of the aluminium ion to induce crystallographical and chemical modifications in a silica backbone (Davidovits 2003). A class of inorganic polymers, geopolymers are formed by reaction between an alkaline solution and an aluminosilicate source (e.g., RHA, fly ash, and slag).

The chemical process to produce geopolymers involve three steps:

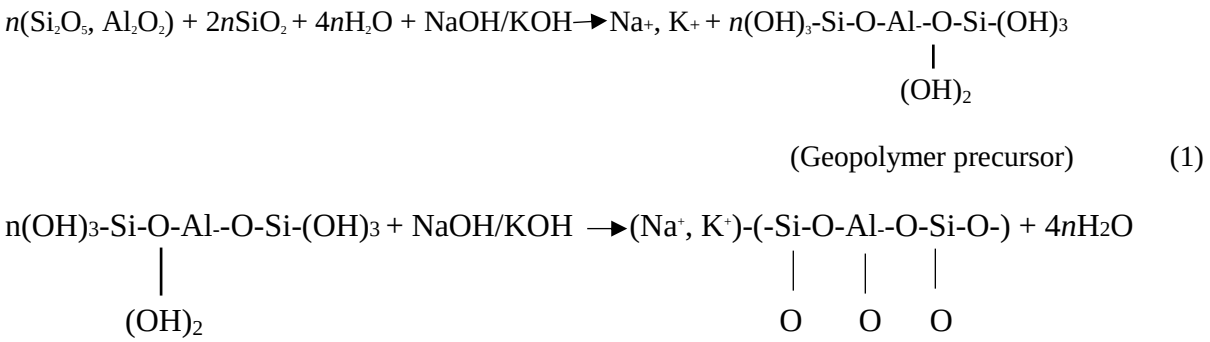
(1) Dissolution of raw materials in alkaline solution to form Si and Al gel on the material's surface,
(2) Reorientation, which is condensation of precursor ions into oligomers
(3) Polycondensation to form networked polymeric oxide structures.

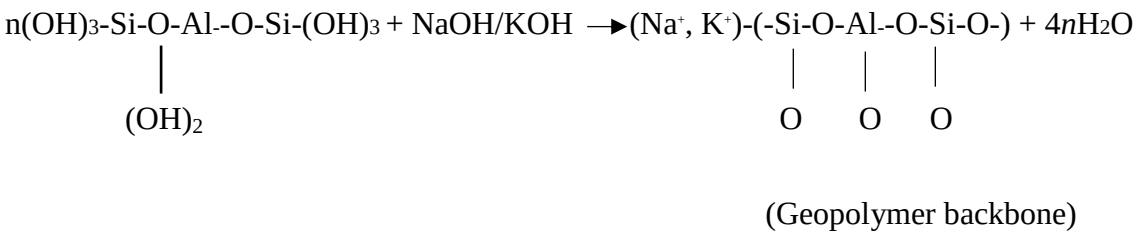
The empirical formula of geopolymer is as follows.

$$M^{+}_{n}\{-(SiO_2)_z{}^{-}AlO_2{}^{-}\}_n$$

Where M^+ = an alkali cation (K^+, Na^+) for balancing the negative charge of $Al_3{}^+$ in IV-fold coordination; n = degree of polymerization; and z = Si/Al ratio.

The schematic formation of geopolymer material is demonstrated by the following two reactions (Xu and Van Deventer 2000):





The above mentioned reactions show that any material mainly containing silica and alumina in glassy phase is a potential source for the making of geopolymers. So far fly ash, GGBFS, RHA, metakaolin and red mud are used as aluminosilicate source. However, the exact mechanism of geopolymerization is not fully interpreted. This new technology is catching attention of researchers as a large amount of waste products can be utilized so that environment pollution can be reduced. There are a lot of research works on geopolymerization which are mainly related to concrete technologies.

Khale and Chaudhary (2007) reported that a source material containing high amount of silica and alumina with an alkali activator is the primary requirement for geopolymerization. The main objective of this research work is the stabilization of four by-products namely fly ash (FA), ground granulated blast furnace slag (GGBFS), rice husk ash

(RHA) and red mud (RM) with different percentages of NaOH (i.e. 5%, 10%, 15%) by investigating the mechanical properties and mineralogical analysis.

Chapter 2
Literature review

Inorganic polymers can be produced by geopolymerization; a progressive technology that can alter various aluminosilicate by-products into beneficial construction materials. Geopolymerization consists of a diverse chemical reaction between aluminosilicate sources and alkali metal ions at highly alkaline conditions and mild temperatures, resulting glassy to semi-crystalline polymeric structures, which consist of Si–O–Al and Si–O–Si bonds [Davidovits, 1999]. Even though mechanism of geopolymerization is not fully understood, the most common method comprises three parallel steps:

(a) Dissolution of solid aluminosilicate materials in alkaline sodium silicate solution

(b) Oligomerization of Si and/or Si–Al in aqueous phase

(c) Solidification of the oligomeric species.

According to Davidovits (1991) geopolymers are comprised of several fundamental poly(sialates) units as shown Fig. 1.

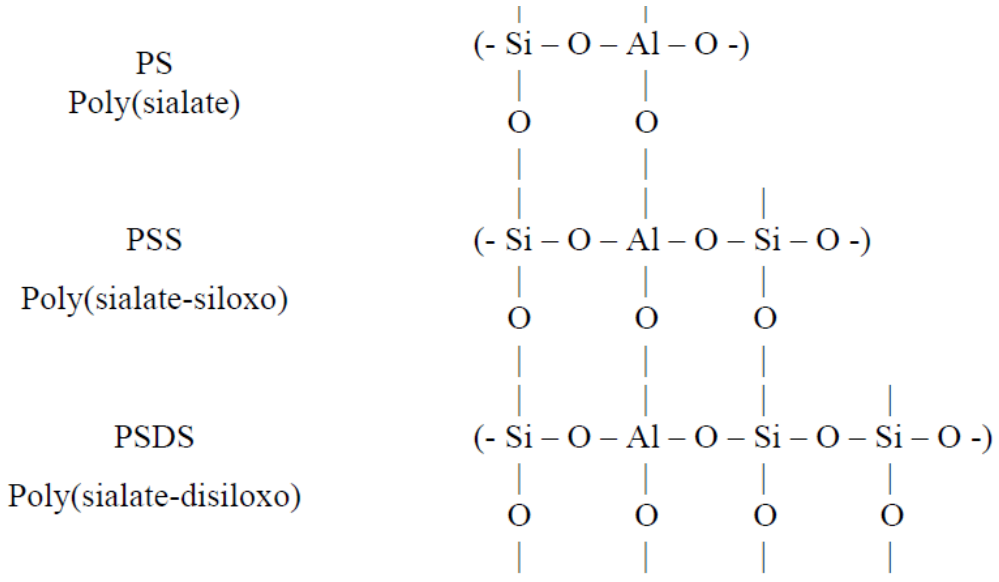


Figure 1 Geopolymeric molecular networks

Katz *(1997)* studied about the activation mechanism of fly ash by an alkaline solution and experiments development of mechanical strength. They analyzed the microstructure of developed products after activation. The conclusion was that increase in the reactivity of material with the increase in concentration of the solution. Curing at elevated temperature is favorable for the activation of fly ash. Higher degree of reaction occurs with higher water/FA ratio.

Palomo *et al***.** *(1998)* stated that the type of activator used in the geopolymerization of the fly ash is a key factor in the progress of reactions. Activators containing soluble silicates induces higher rate of reaction and higher strength production than the only hydroxide solution.

Puertas *et al.* (2000) described the behavior of fly ash and slag mixture with different proportion (100/0, 70/30, 50/50, 30/70 and 0/100) after alkali activation with two concentrations of NaOH (2M and 10M) and they kept the samples at two curing temperature i.e. 25^{o}C and 65^{o}C The conclusion extracted from the research work was (i) As slag content increases, compressive strength increase (ii) Concentration of NaOH is the primary ingredient in the energizing process. At higher concentration, i.e. 10M, higher strengths are obtained. In the starting days of reaction, the curing temperature has a positive effect and with curing age, the effect is opposite and strength at 25^{o}C gives better result than 65^{o}C.

Jaarsveld *et al.*(2002) investigate the use of geopolymers to immobilize the toxic materials. In their research work, raw materials were fly ash and kaolin. They concluded that the final properties of the geopolymer depends on the fly ash/kaolinite ratio, water content as well as the type of metal.

Cheng and Chiu (2003) tried to fabricate a GGBFS based geopolymer to use in fire resistance purpose. KOH and sodium silicate were used as alkali activator in their research work. GGBFS and metakaolin were taken as raw materials. The samples were cured at 60^{o}C for 3 hours in an oven and then kept at room temperature for 21 hours before beginning of

experiments. With increase in KOH concentration, strength of specimen increases. For 10N KOH concentration, the strength was achieved maximum of 79 MPa and for concentration more than 10N, strength got reduced. They also found that with increase in addition of metakaolin, strength increases that is may be due to more availability of $Al3^{+}$ ions for geopolymeric reaction.

Puertas *et al.* (2003) observed strength, mineralogical and microstructural analysis of mortar (comprising activated fly ash/slag mixtures) after cured at different temperatures. Characterization of product is done by X-Ray Diffraction, FTIR, MAS-NMR, Scanning Electron Microscope, EDX, atomic absorption and chromatography. The products found are calcium silicate hydrate rich in aluminium and alkaline aluminium silicate hydrate.

Fernandez-Jimenez *et al.* (2005) conducted a study on mechanical strength of alkali activated fly ash mortars with different type of activator. The behavior and microstructure of reaction products were observed. The primary reaction product after activating fly ash was an alkaline aluminosilicate gel. Hydroxyl ion acts as a reaction promoter throughout the geopolymerization, and alkali metal acts as a structure making component.

Criado *et al*. (2007) studied the effect of soluble silica on the microstructural development of ash activator reaction and its effect on mechanical development of material. They concluded major reaction product sodium aluminosilicate gel gives good strength to raw materials. Zeolites are formed as minor products in the reaction. The amount of zeolites rises with curing time for curing temperature of $85^{o}C$.

Kong *et al.* (2008) demonstrated a study on fire resistance of geopolymers/geopolymer aggregate composites by keeping samples at elevated temperature (up to $800^{o}C$). It resulted as a hike in strength of geopolymer after curing at higher temperature. Both polymerization of silica and alumina precursor and sintering at higher temperature gave better result for

strength development whereas strength decreased after exposure to temperature due to differential thermal expansion between geopolymer and aggregates.

Zhang *et al.* (2009) experimented on the possibility of incorporating red mud and coal gangue as raw material for the cementitious material. The physical and mechanical properties show the usefulness. The hydration products of RGC are mostly ettringite and CSH gel.

The probable utilization of red mud in the production of geopolymeric materials was investigated by **Dimas** *et al.* (2009). The influence of primary synthesis factors like – solid/liquid, sodium hydroxide, sodium silicate concentrations and percentage of metakaolin added controlled the behaviour of synthesizing material. The optimum conditions for synthesis were concluded as a mixture of 85% red mud and 15% metakaolin with an aqueous solution (consisting of $[SiO_2]$ = 3.5M, [NaOH] = 8M) at S/L ratio=2. 9 g/ml).

Heah *et al.* (2011) studied on kaolin based geopolymer and observed the influence of curing temperature on the geopolymer. The conclusion of the research work are: (a) Curing at ambient temperature is not possible for the prepared geopolymer due to delays in the beginning of setting time. (b) Disintegration of reactive species and development of higher strength is favored by elevation in temperature. (c) Maximum development strength was achieved at temperature of 60^oC when cured for 3 days and the microstructure also supported the results by showing more geopolymeric gel in the samples.

Mustafa *et al.* (2011) experimented on fly ash based geopolymer samples. They chose mixture of sodium silicate and sodium hydroxide as alkaline activator. Various temperatures (room temperature, 50^oC, 60^oC 70^oC, 80^oC) were chosen for curing with the duration of 24 hour. The 7^{th} day strength revealed that maximum compressive strength of 67.04MPa is found for the samples cured at temperature of 60^oC.

Arioz *et al.* (2012) took fly ash based geopolymer for the research work. They investigated mechanical and microstructural properties of the prepared materials which was activated by 4M, 8M and 12M of sodium hydroxide and sodium silicate solutions. All the specimens were cured at 80^{o}C for 15hours. On 7^{th} day and 28^{th} day, strength tests were carried out. The microstructure of the samples was examined by Scanning Electron Microscope, EDX, X-Ray Diffraction and FTIR. They concluded that with the increase in concentration of sodium hydroxide solution the compressive strength and degree of reaction increases.

Zhang *et al.* (2013) examined the viability of geopolymer in stabilizing lean clay. Metakaolin based geopolymer was used for the stabilization lean clay with varying concentration at OMC. The concentration ranges from 3 to 15 weight percentage of unstabilized soil. Specimens are characterized for compressive strength tests, Scanning Electron Microscope, EDX, FTIR, X-Ray Diffraction, volume change during curing.

The research work available on the effects of using NaOH alone as the alkali activator for the development of geopolymer materials is limited. The present research aims to explore the activation of by-products with different percentages of NaOH. Strength characteristics of the activated materials were observed by unconfined compressive test (UCS test). XRD analysis was carried out to find out the minerals produced after the alkali activation.

Chapter 3
Material and methodology

3.1 Materials used:

3.1.1 Ground Granulated blast furnace slag*:*

Ground Granulated Blast Furnace Slag (GGBFS) is defined as the amorphous product comprising basically of calcium silicates and other bases. The major components of blast-furnace slag are silica, alumina, calcium oxide, and magnesia. Typical chemical composition is given in Table 1. Therefore GGBFS can be used as aluminosilicate source. For this research work, GGBFS was collected from Rourkela steel plant. The material was dried in oven to remove the water present in raw material and put in a ball mill to increase the fineness. The material sieved through 75 μm was used for the project work.

Table 1: Typical chemical composition of GGBFS

Mineral name	Calcium oxide	Silica	Alumina	Magnesia
percentage	40%	35%	13%	8%

3.1.2 Fly ash:

Fly ash is aluminosilicate source as main compositions of the fly ash are CaO, SiO_2, and Al_2O_3. For this project work, fly ash is collected from Adhunik Metaliks Limited, Sudergarh. The materials was oven dried and kept in a safe place for future use.

3.1.3 Rice husk ash:

RHA contains a very high amount of silica content and is found to be amorphous in nature. The amorphous silica contained in RHA can react with cementitious binders to perform pozzolanic activity. Locally available rice husk ash was used to carry out the research work. In this research work, dry RHA were sieved to remove residual bran and clay particles and kept in oven at 105^oC-110^oC for 24 hours before sample preparation.

3.1.4 Red mud:

Red Mud is the insoluble product after bauxite digestion with sodium hydroxide at higher temperature and pressure. It is a combination of minerals initially available in the raw ore, bauxite, and of compounds introduced during the Bayer cycle. Wet disposal of red mud is mostly preferred by preparing a slurry with 10-30% of solid concentration. The pH of red mud is generally in the range of 11-13. The amount of red mud generated gets influenced by the quality of bauxite ore and it varies from 55-65% of the treated bauxite. Alkalinity of red mud in spite of a high water content, is high because of the existence of an extreme amount of dissolved sodium hydroxide. Moreover, constituents of red mud comprise generally iron oxides, alumina, and silica. Red mud was collected from NALCO, Damanjodi. The material was oven dried and the fineness of the material was increased by putting it in a ball mill. The red mud use in this project work had pH of 11.2.

3.1.5 Sodium hydroxide:

Sodium hydroxide was used for the alkali activation of the raw materials (i.e. Slag, fly ash, rice husk ash, red mud). The sodium hydroxide pellets used for this project were Fisher Scientific brand with 98% purity. The NaOH solution was prepared before 24 hours to ensure proper dissolution of the sodium hydroxide pellets.

3.2 Methodology

3.2.1 Specific gravity:-

The specific gravity of fly ash was determined according to IS: 2720 (Part-III, section-1) 1980 by using pycnometer with distilled water as the solvent. The values are shown in Table 2.

Table 2: Specific gravity of the raw materials

Material	Specific gravity
Slag	2.75
Fly ash	2.59
Rice husk ash	2.01
Red mud	3.13

3.2.2 Moisture Content-Dry Density Relationship:

The compaction characteristics of the materials were determined as per IS 2720 (Part VII) 1980. Four materials were activated by varying percentage of NaOH (i.e. 5%, 10%, 15%) of its drying weight. The sodium hydroxide solution was prepared with the starting percentage of water to be added to dry sample for standard proctor test. The solution was kept for 24 hrs. to dissolve the sodium hydroxide pallets properly. As per IS: 2720 (Part II) 1973 the moisture content of the compacted mixture was determined. From the dry density and moisture content relationship, optimum moisture content (OMC) and maximum dry density (MDD) were determined.

3.2.3 Unconfined compressive strength:

For determination of strength of alkali activated raw material at different curing days (0day, 7days and 28 days), 100mm height and 50 mm diameter samples are prepared at OMC and MDD by adding varying percentage of NaOH with oven dried raw material. Unconfined compressive strength tests were conducted as per IS 2720 (Part X) 1991. The samples were coated with wax and kept for curing at ambient temperature. The UCS tests were conducted on the prefixed curing days. Curing periods for the project work were 0 day, 7 days and 28 days. UCS tests for the determination of immediate strength were conducted within 2hrs after mixing the raw material with alkaline solution.

3.2.4 XRD analysis:

The X-ray diffraction (XRD) tests were used for the determination of the crystalline phase of the minerals present in the raw materials and activated samples. XRD test is performed by using Philips X' PERT System X-Ray diffractometer. After prefixed curing period, samples were collected and soaked in acetone to discontinue the ongoing reaction. Samples were made finer than 75 µm before taking it for XRD analysis. The specimen was positioned in the diffractometer and scanned using Cu Kα radiation, a step size of 0.02˚ with range 10^o to 70^o at a rate of 10^o/min. XRD Pattern of all the four by-products are shown in fig. (2)-(5). From XRD analysis, it was observed that quartz (SiO_2) is present in all raw materials. A hump between 2 theta (20^o -40^o) is observed from XRD pattern of GGBFS which represents the glassy structure of GGBFS. From XRD analysis of fly ash, the crystalline peaks are found to be hematite (Fe_2O_3) and quartz (SiO_2). From fig 4, we can conclude that hematite, magnetite, quartz and aluminium oxide are the minerals present in red mud. The RHA contains silica and cristobalite and the halo in the range of 10^o-30^o represents the amorphous structure of RHA.

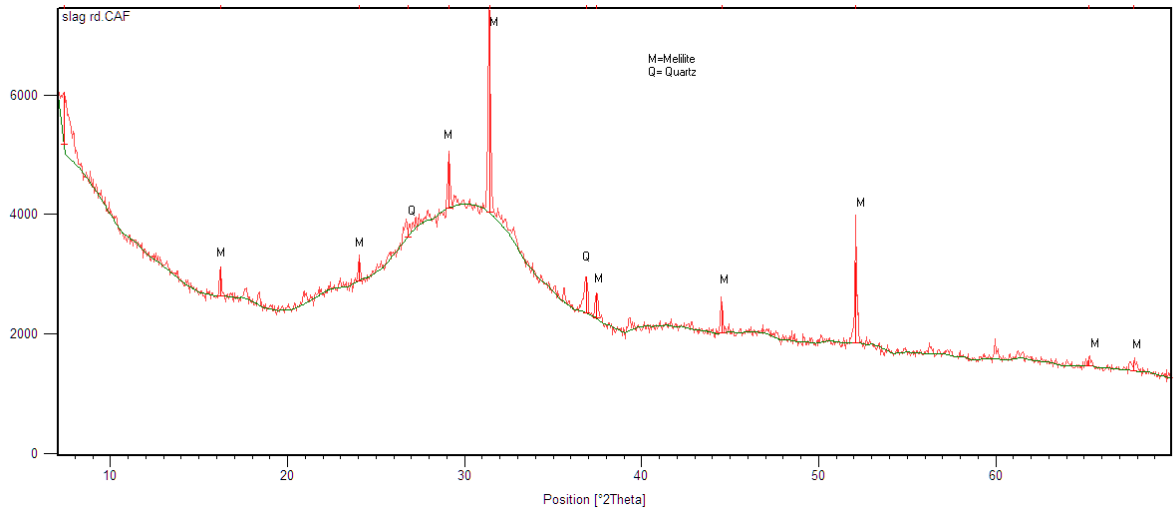


Figure 2: XRD pattern of GGBFS

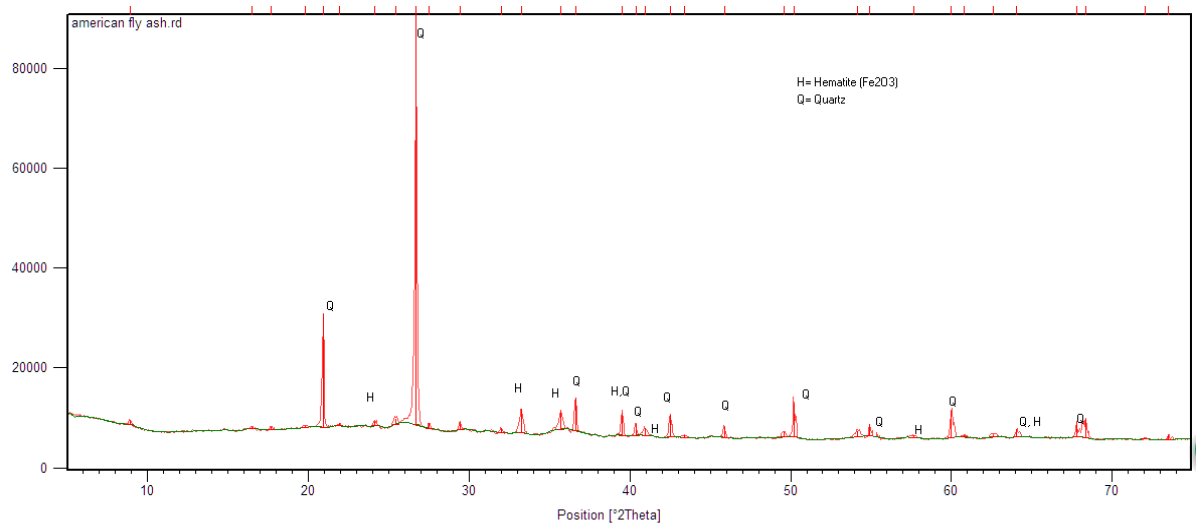


Figure 3: XRD Pattern of Fly Ash

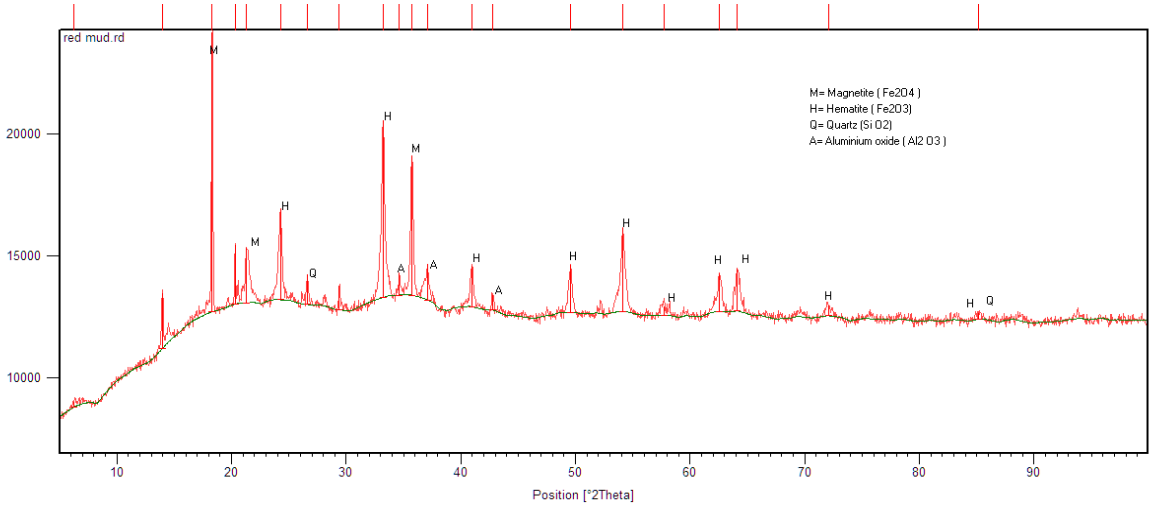


Figure 4: XRD Pattern of Red mud

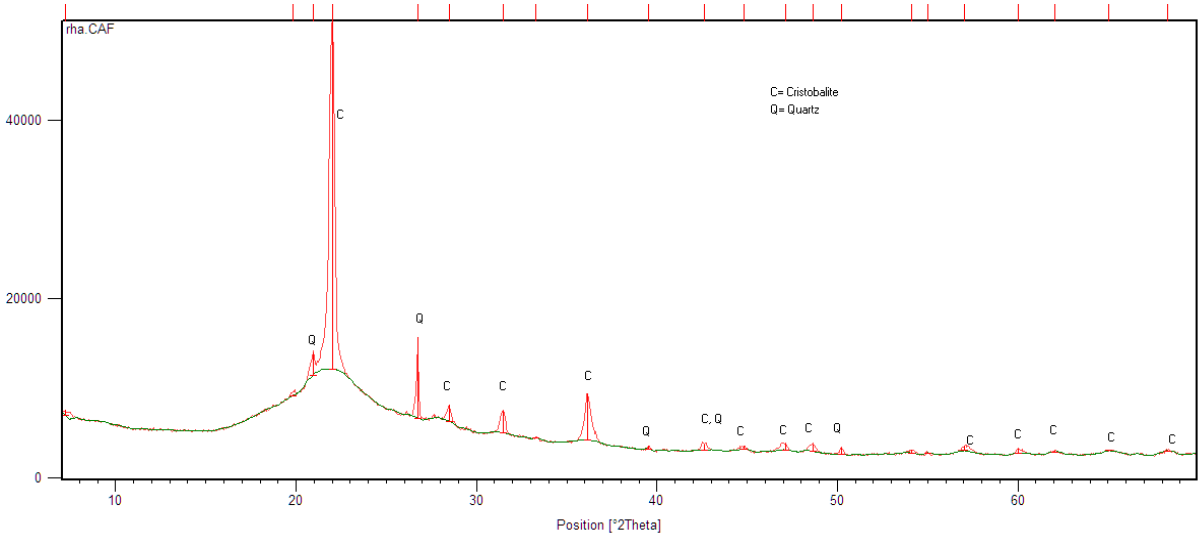


Figure 5: XRD pattern of rice husk ash

Chapter 4
Result and discussion

4.1 Light Compaction Test:

The Fig. (1)- Fig (4) represent the compaction characteristics of slag, fly ash, red mud and rice husk ash with different percentage of NaOH (i.e. 0%, 5%, 10%, 15%) respectively. With increase in percentage of NaOH added to raw material, optimum moisture content (OMC) decreases and maximum dry density (MDD) increases.

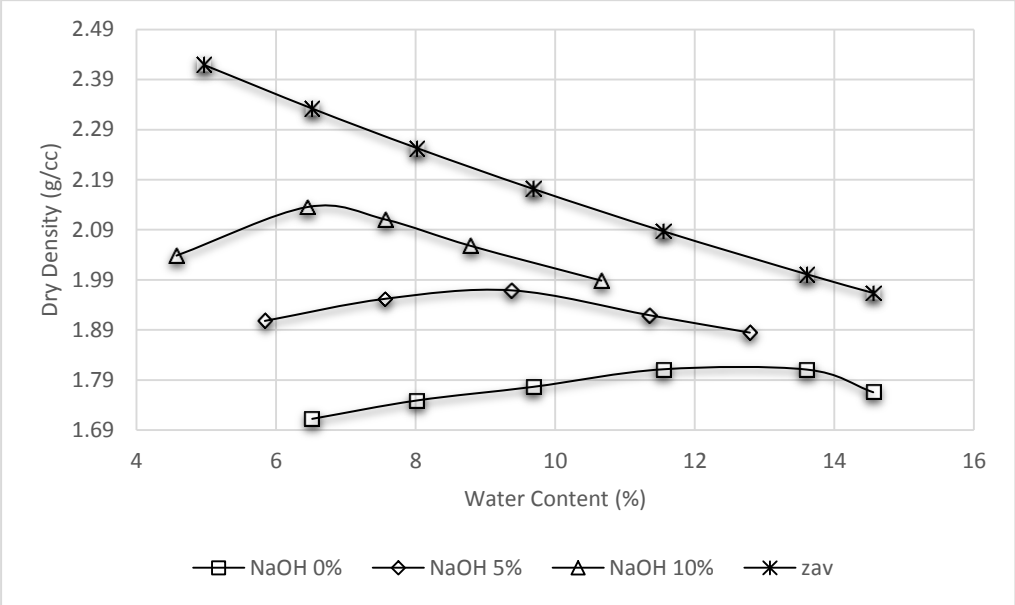


Figure 6 Moisture content-dry density relationship for slag with different % of alkali content

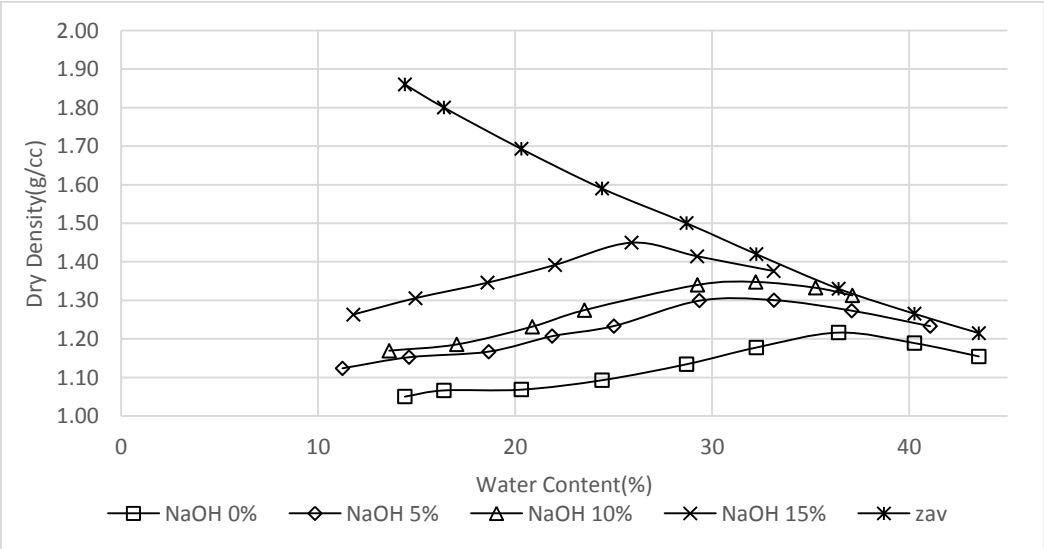


Figure 7: Moisture content- dry density relationship of fly ash at different alkali content

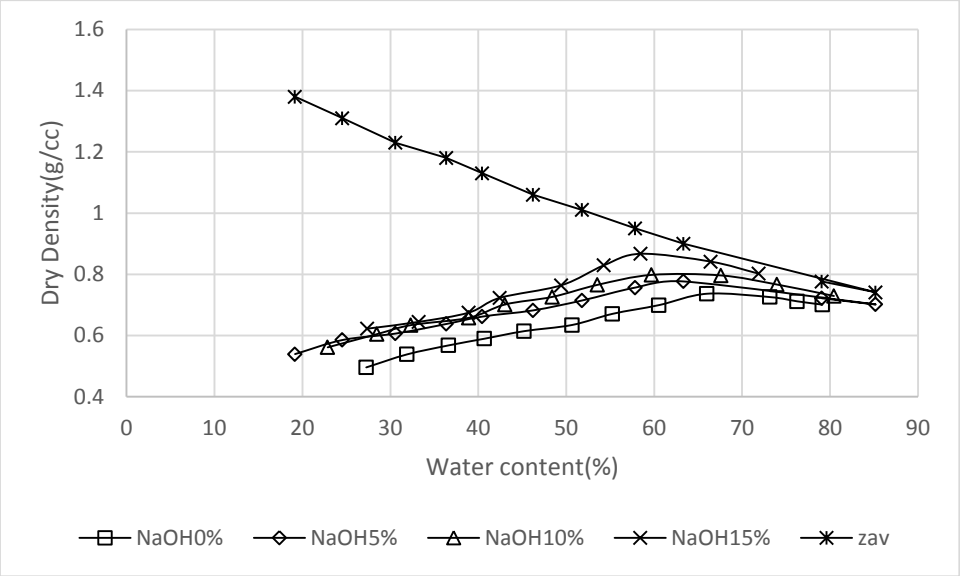


Figure 8: Moisture content- dry density relationship for rice husk ash with different % of alkali content

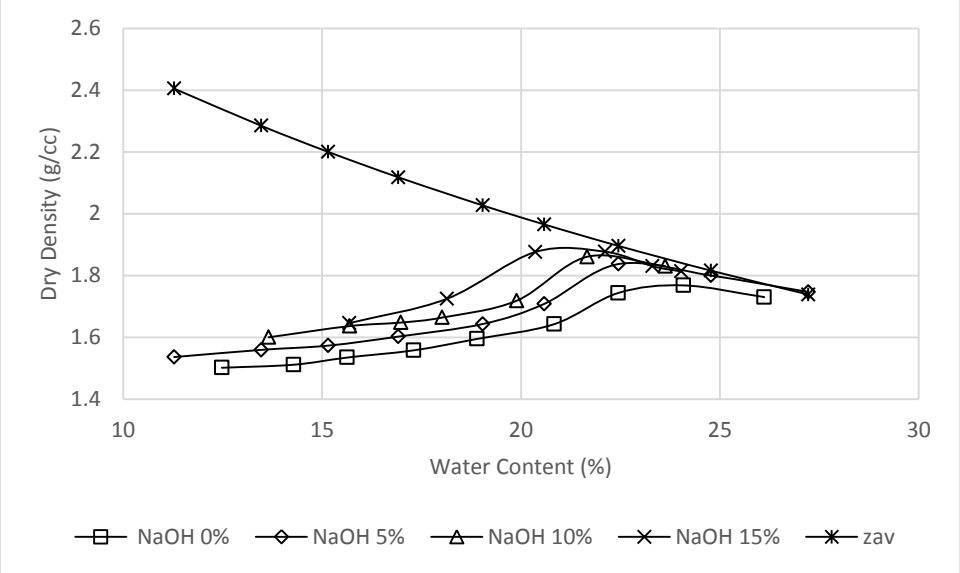


Figure 9: Moisture content- dry density relationship for Red Mud with different % of alkali content

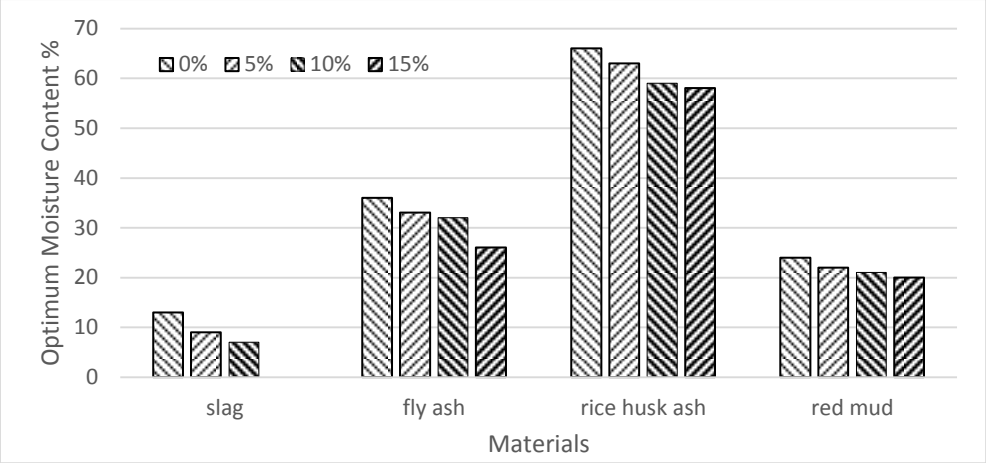


Figure 10: Variation of OMC with different percentages of NaOH

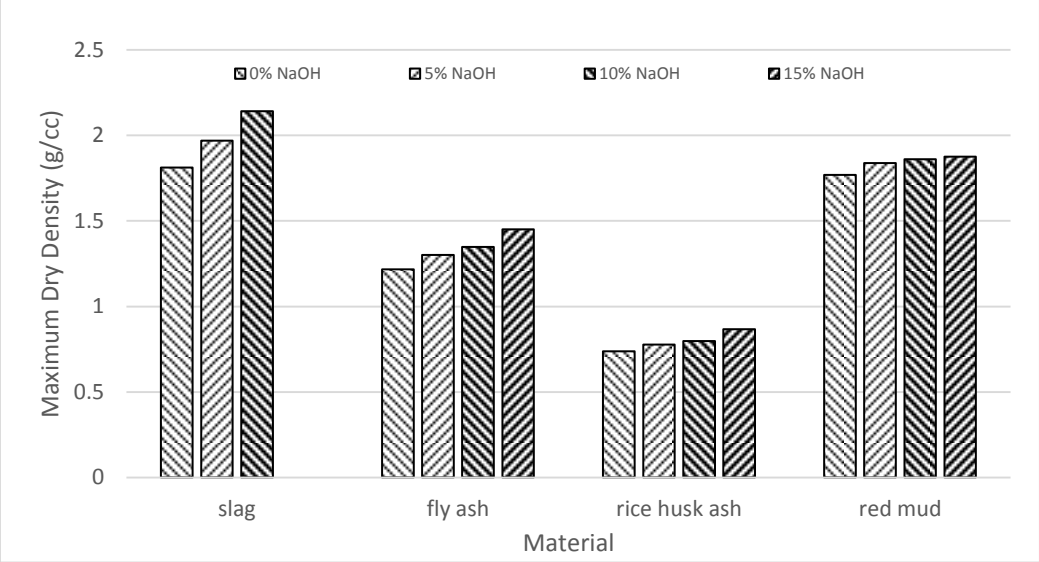


Figure 11: Variation of MDD with different percentages of NaOH

Table 3: OMC and MDD of raw materials with varying NaOH %

Description	Optimum moisture content (%)	Maximum dry density(g/cc)
Fly ash	36	1.216
Fly ash+5% NaOH	33	1.301
Fly ash+10% NaOH	32	1.347
Fly ash+15% NaOH	26	1.45
Slag	13	1.811
Slag+5% NaOH	9	1.969
Slag+ 10% NaOH	7	2.14
Red mud	24	1.768
Red mud+5% NaOH	22	1.837
Red mud+10% NaOH	21	1.86
Red mud+15% NaOH	20	1.876
RHA	66	0.737
RHA+ 5% NaOH	63	0.777
RHA+ 10% NaOH	59	0.798
RHA+ 15% NaOH	58	0.867

4.2 Unconfined strength test:

4.2.1 Slag

As shown in Fig 8, the immediate strength of slag with 5% NaOH is 2.89 times more than the strength given by the slag without any activation whereas the strength of slag sample with 10% NaOH gives 7.6 times more than raw sample**.**

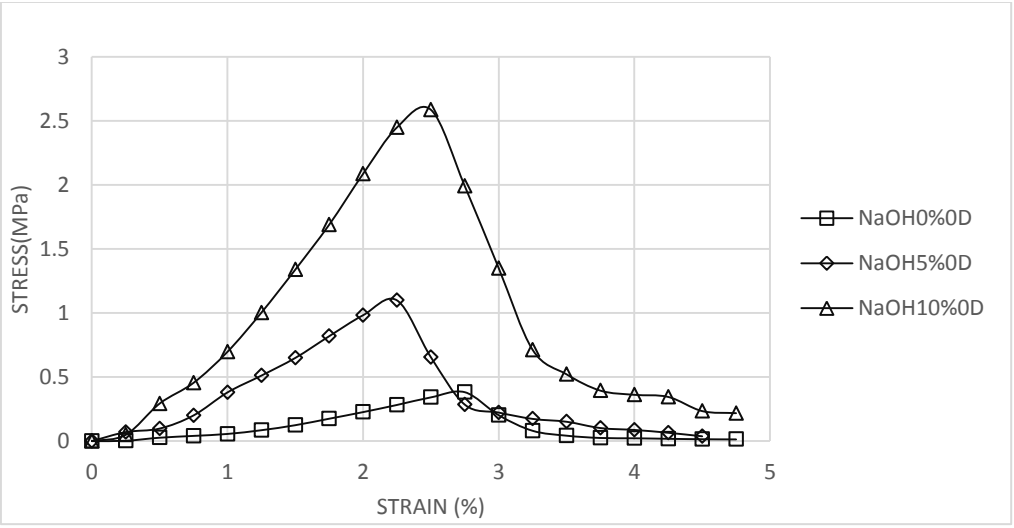


Figure 12: Stress- Strain curve of slag with different % NaOH at 0day

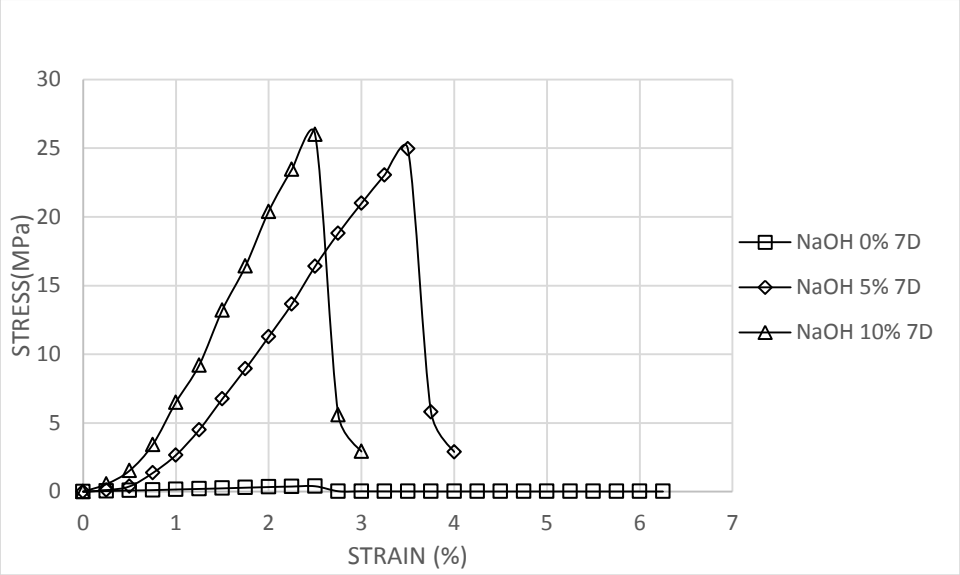


Figure 13: Stress- Strain curve of slag with different % NaOH at 7days

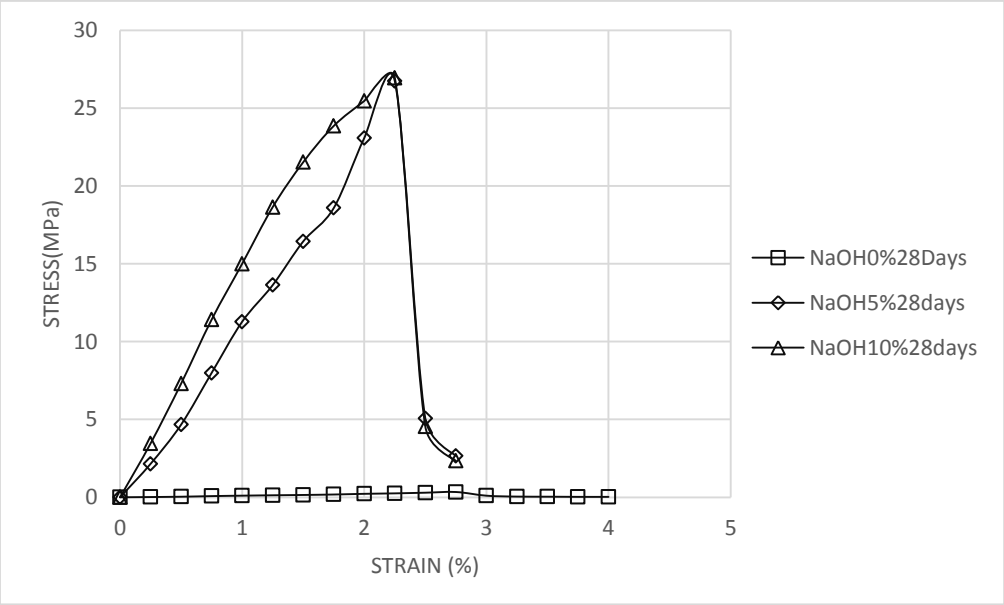


Figure 14: Stress- Strain curve of slag with different % NaOH at 28days

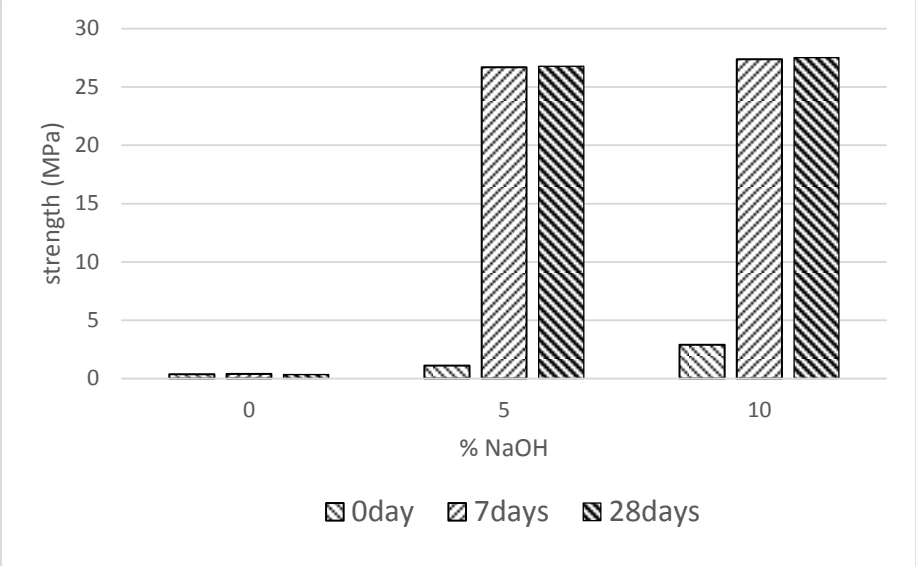


Figure 15: Comparison of strength of slag at different curing periods

Fig. (10) and (11) show the stress- strain relationship of the activated sample at 7 days and 28 days curinge respectively. From those figures, it can be concluded that samples becomes more brittle with curing periods. At 7 days, the strength of samples with 5% NaOH and 10% NaOH are 26.66 MPa and 27.37 MPa respectively which are almost 70 folds of the strength given by the specimens having no chemicals. At 28 days, there is no significance changes in the strength. As per fig. (12), conclusion can be drawn that there was no significance change in development of strength at 28 days curing period.

4.2.2 Fly ash:

Fig 12 shows the immediate strength of fly ash with different percentages of NaOH (5%, 10% and 15%). Strength of samples with 5% NaOH is .55 MPa, which is 2 time more than the strength of raw samples.

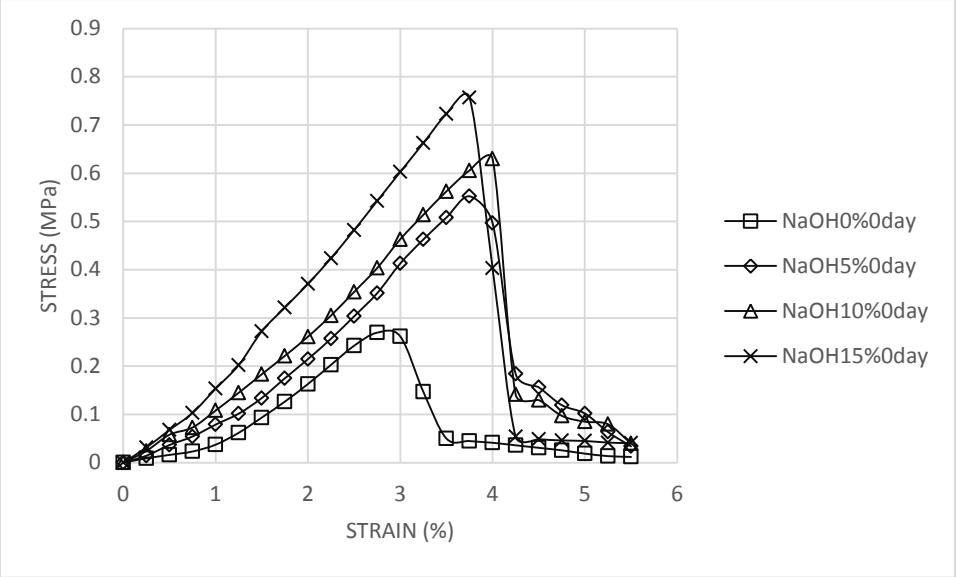


Figure 16: Stress- Strain curve of Fly ash with different % NaOH at 0day curing

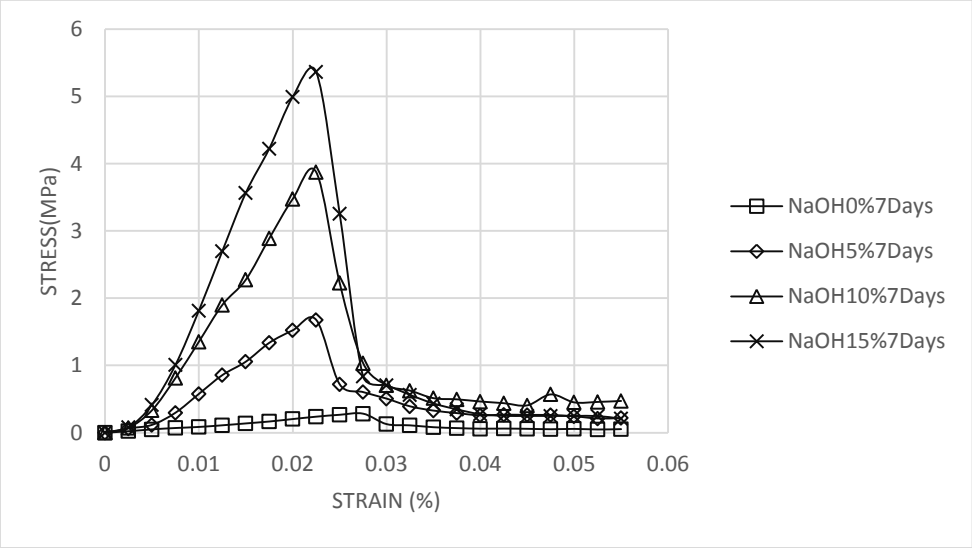


Figure 17: Stress- Strain curve of Fly ash with different % NaOH at 7days curing

With increase in curing period, the strength of activated samples increase approximately 20 times compared to raw material. At 28 days curing, for 10% and 15% NaOH samples, the gain in strength is almost same.

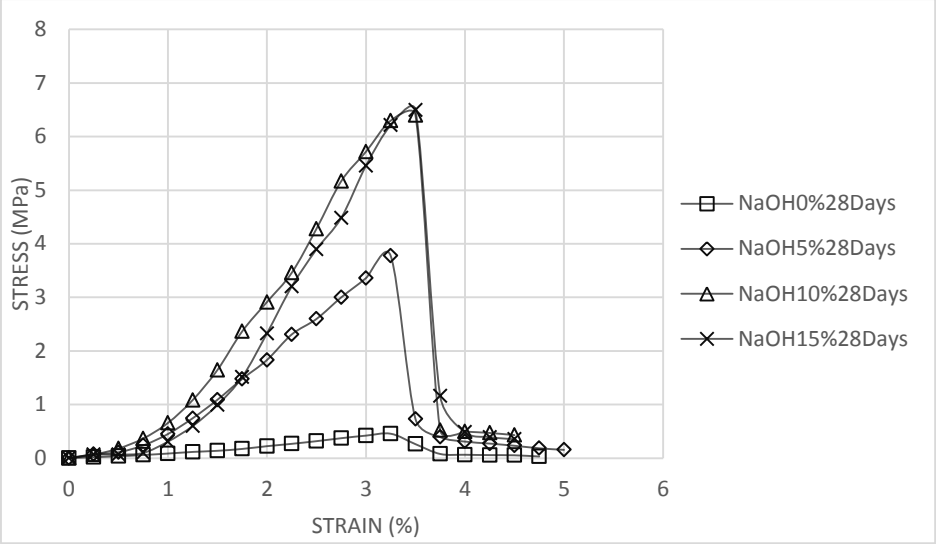


Figure 18: Stress- Strain curve of Fly ash with different % NaOH at 28days curing

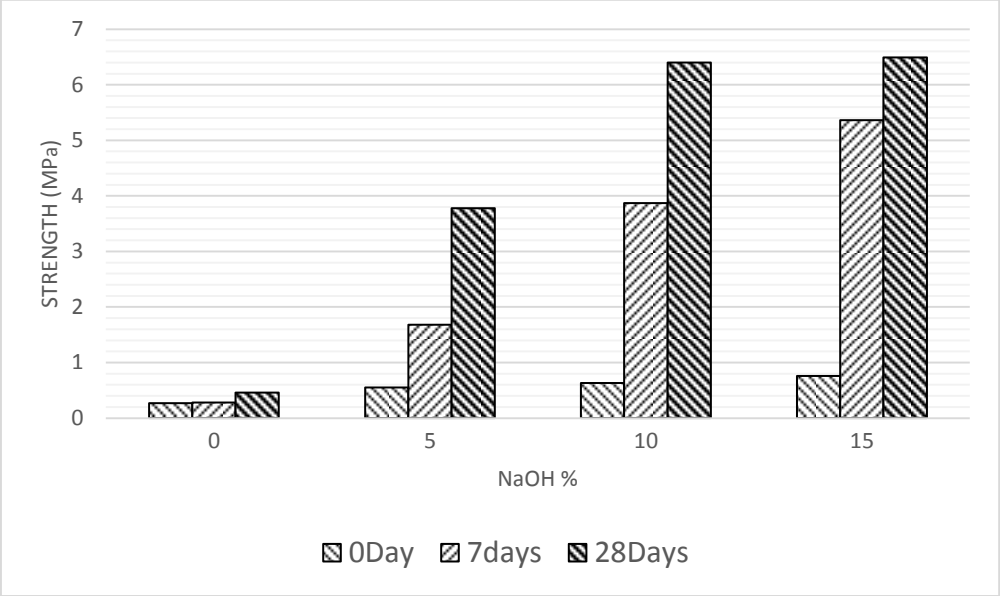


Figure 19: Comparison of strength of Fly ash at different curing periods

Fig. 15 shows the comparison of strength gain by specimens at different curing days. From the graph it can be concluded that for 7 days curing, the strength gain by 5% NaOH and 10% NaOH activated samples are 6 times and 13 times more than raw material whereas the gain by 15% NaOH activated samples are 20 folds more.

4.2.3 Rice husk ash

In case of rice husk ash, the strength of specimens gets reduced after adding NaOH. Ductile failures occur for higher percentages of NaOH as shown in Fig. (16) – (18). At 28 days curing, the strength of specimen increases as compared to 7 days curing. The specimen gets more bulged during failure.

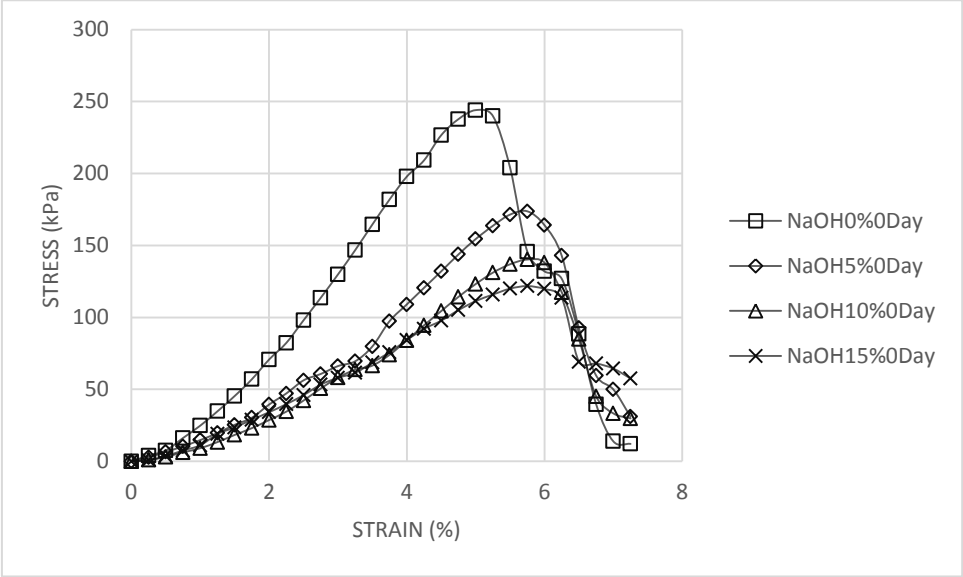


Figure 20: Stress- Strain curve of Rice husk ash with different % NaOH at 0 days curing

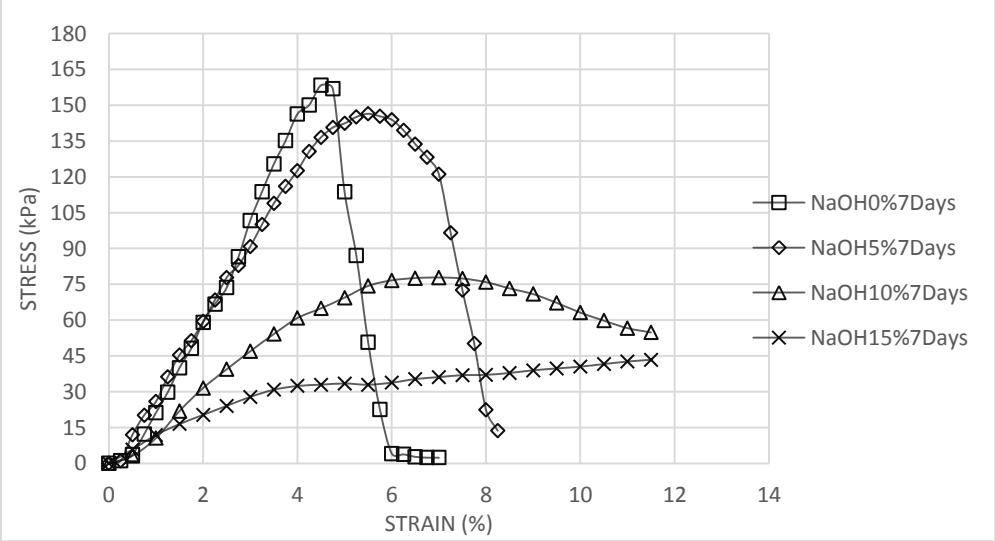


Figure 21: Stress- Strain curve of Rice husk ash with different % NaOH at 7 days curing

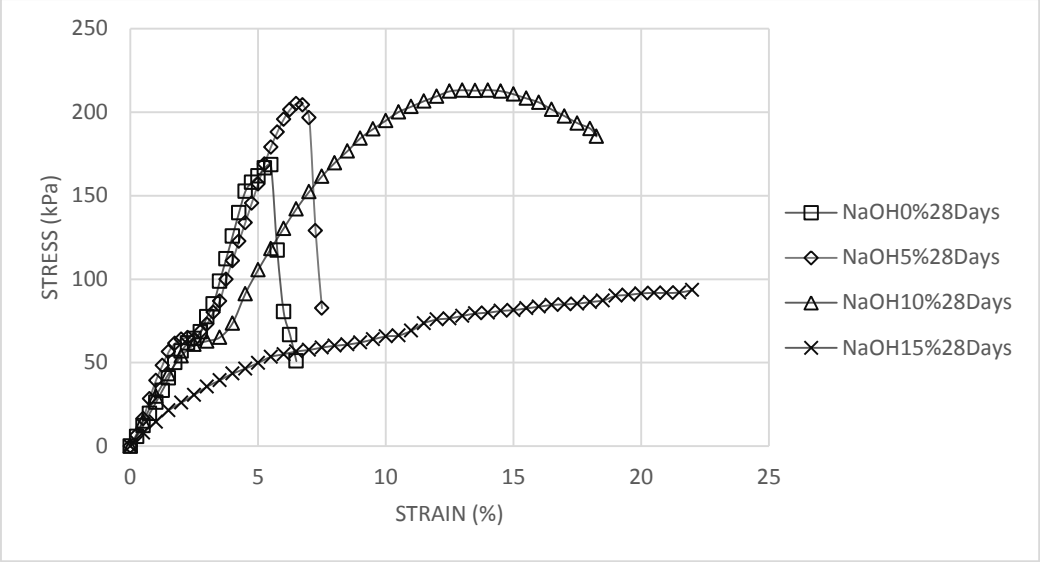


Figure 22: Stress- Strain curve of Rice husk ash with different % NaOH at 28 days curing

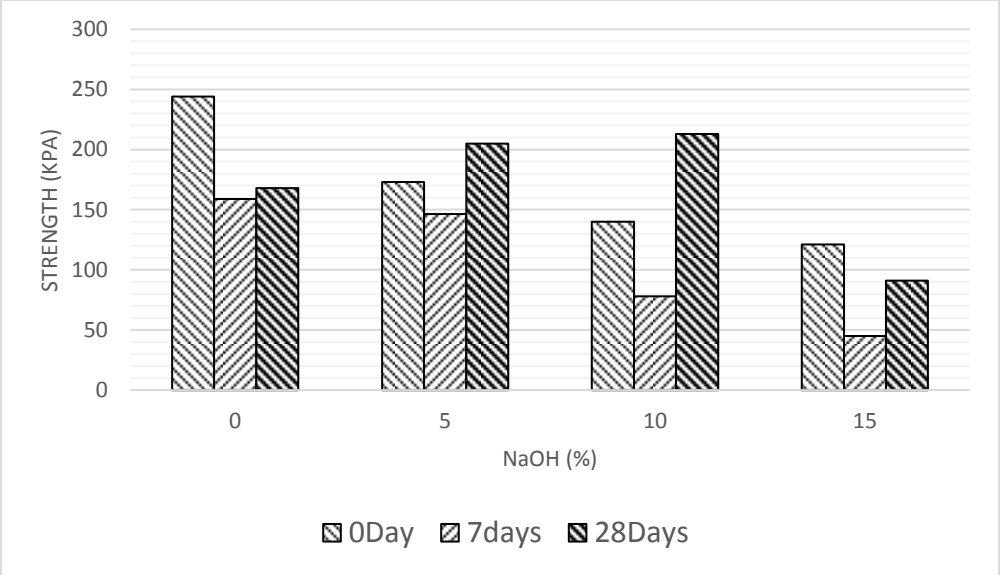


Figure 23: Comparison of strength of Rice husk ash at different curing periods

The change in strength of activated samples with different curing periods is shown in Fig 23. First on the 7th day of curing, the strength of samples decreases but on the 28th day, it increases slightly. With increase in percentage of NaOH, the strength decreases.

4.2.4 Red mud:

In the case of red mud, the immediate strength of activated specimen decreases in comparison to non-activated samples. After 7 days of curing period the strength of sample

with 5% NaOH increases from the non-activated samples, but for other samples, the strength is not significant.

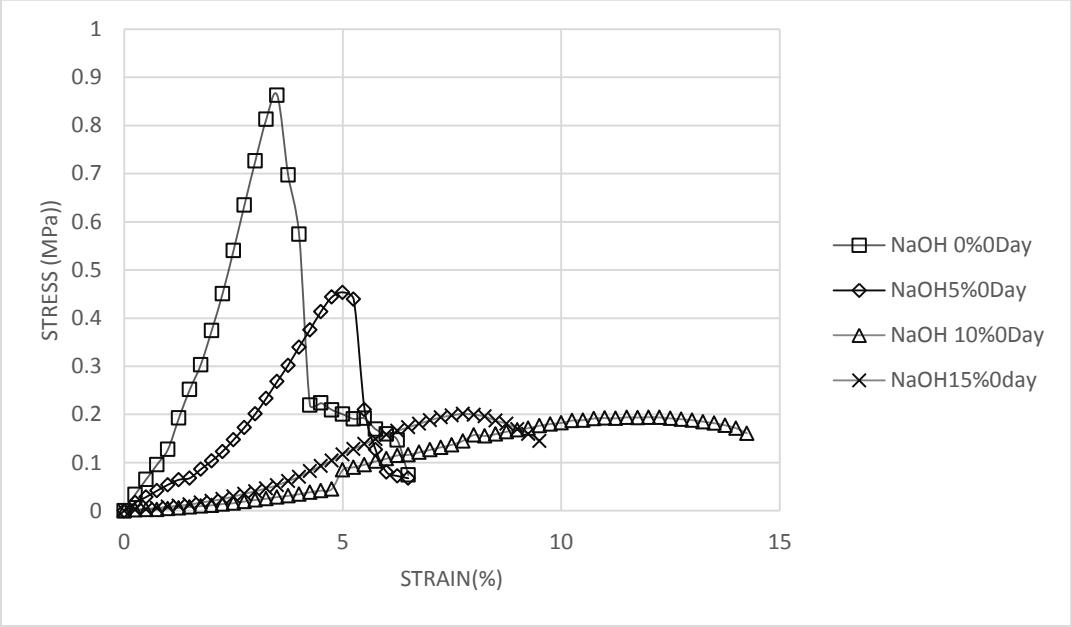


Figure 24: Stress- Strain curve of Red mud with different % NaOH at 0 days curing

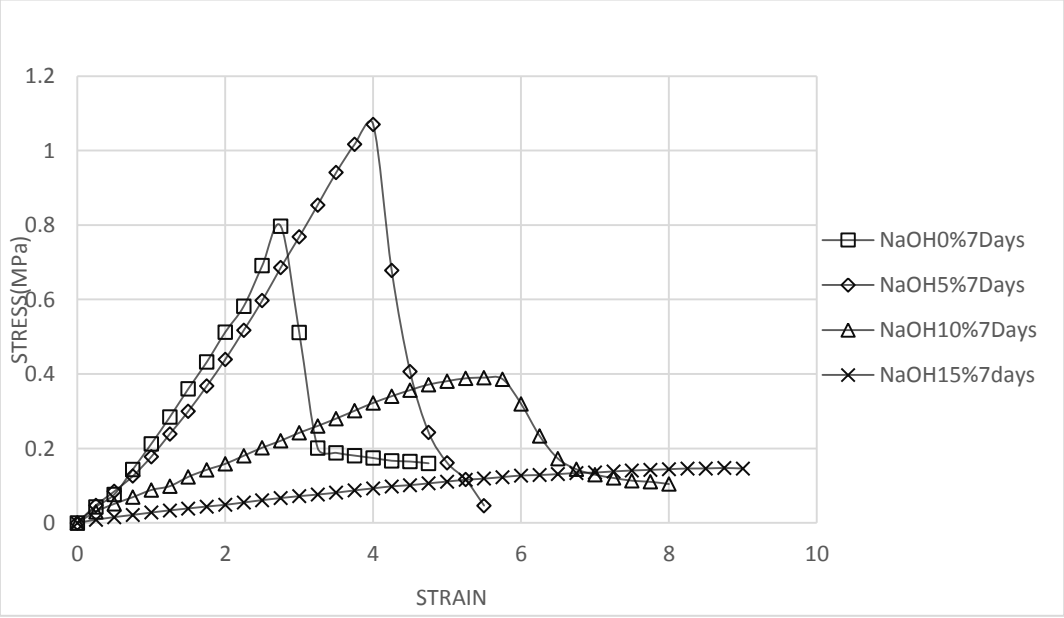


Figure 25: Stress- Strain curve of Red mud with different % NaOH at 7 days curing

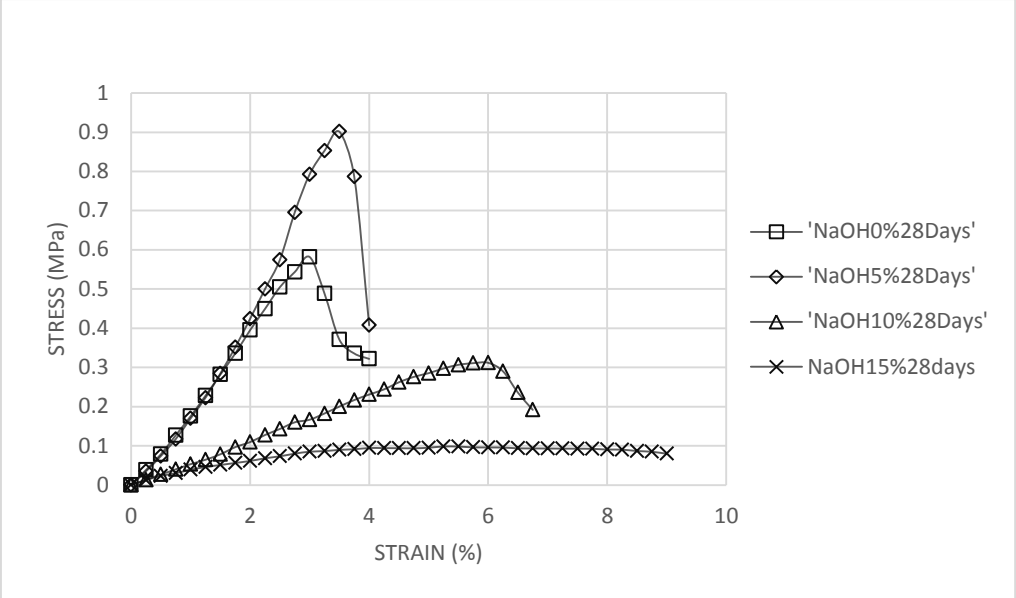


Figure 26: Stress- Strain curve of Red mud with different % NaOH at 28 days curing

With addition of 5% NaOH, the strength of red mud increases to 1.08MPa after 7 days curing. For samples with 15% NaOH, strength decreases with curing periods. The immediate strength of the sample was found to be 0.201MPa while in case of 28 days curing, it came down to 0.146.

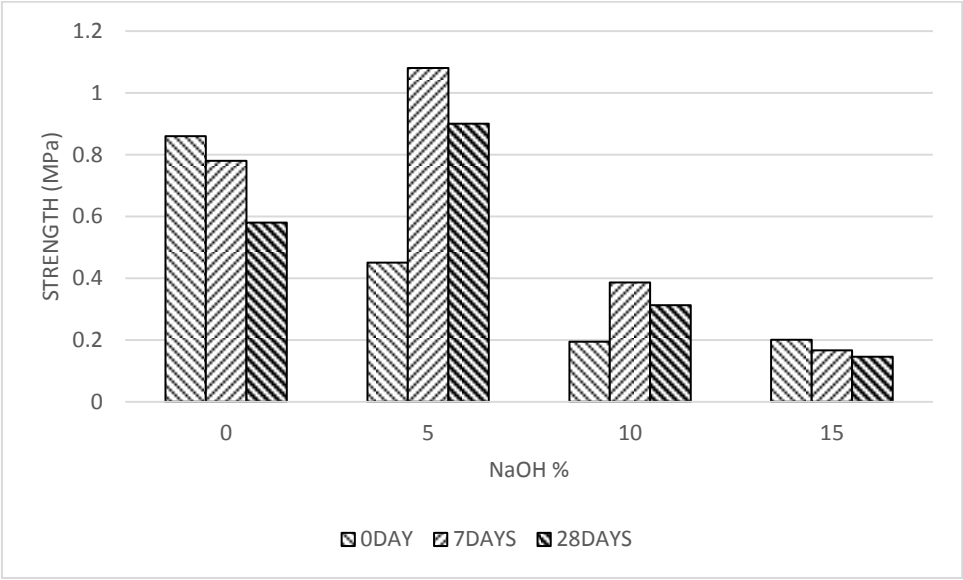


Figure 27: Comparison of strength of red mud at different curing periods

4.3 XRD analysis

Fig 28 shows the XRD pattern of GGBFS and activated GGBFS with 5% NaOH at 7 days. In activated sample, peaks of quartz minerals are not detected. It can be concluded that silica took part in the reaction.

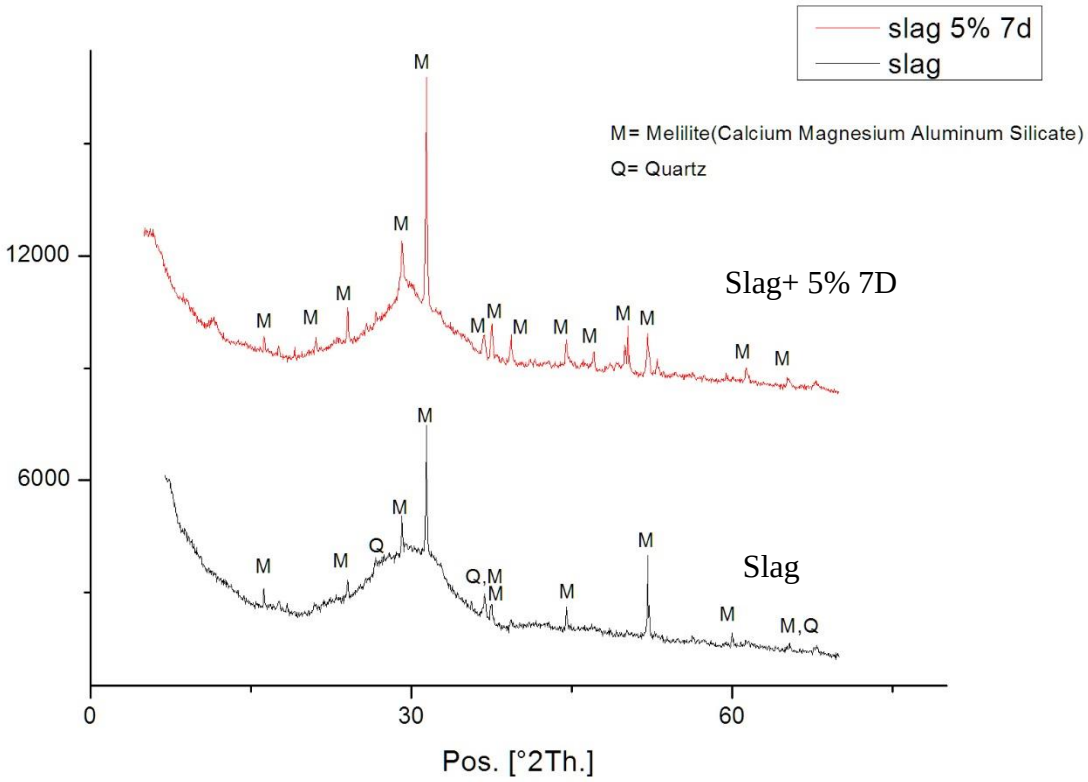


Figure 28: XRD pattern of GGBFS and activated GGBFS (5% NaOH)

Sodium aluminium silicate was detected from the XRD pattern of the red mud with 5% NaOH sample after 7 days of curing which is shown in Fig. 30. From XRD pattern of activated RHA with 5% NaOH, peaks of sodium silicate were observed. Crystalline peaks of Sodium aluminium silicate were obtained from the XRD pattern of alkali activated fly ash depicted in fig. 29. The crystal size of quartz got reduced due to activation of fly ash.

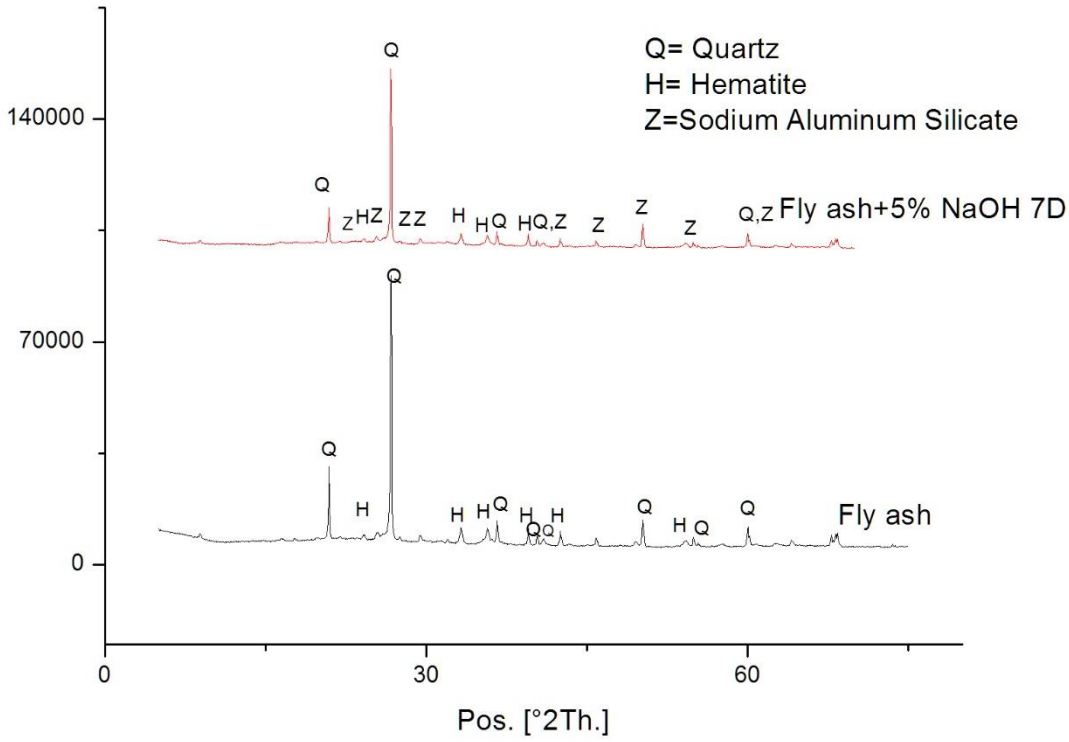


Figure 29: XRD pattern of fly ash and activated fly ash (5% NaOH)

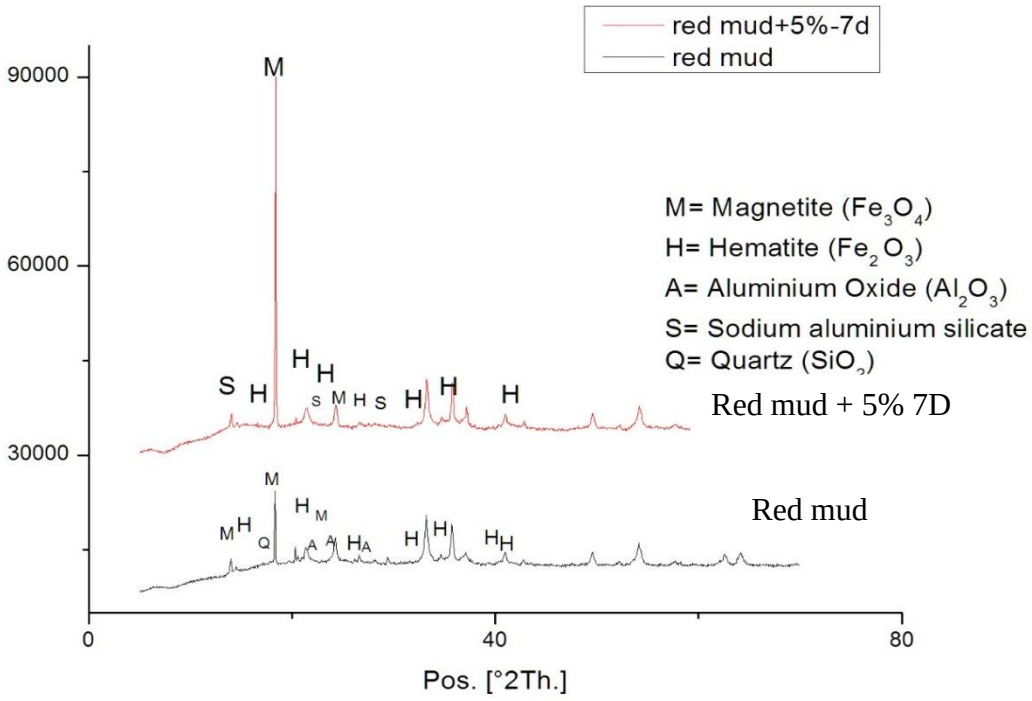


Figure 30: XRD pattern of Red mud and activated Red mud (5% NaOH)

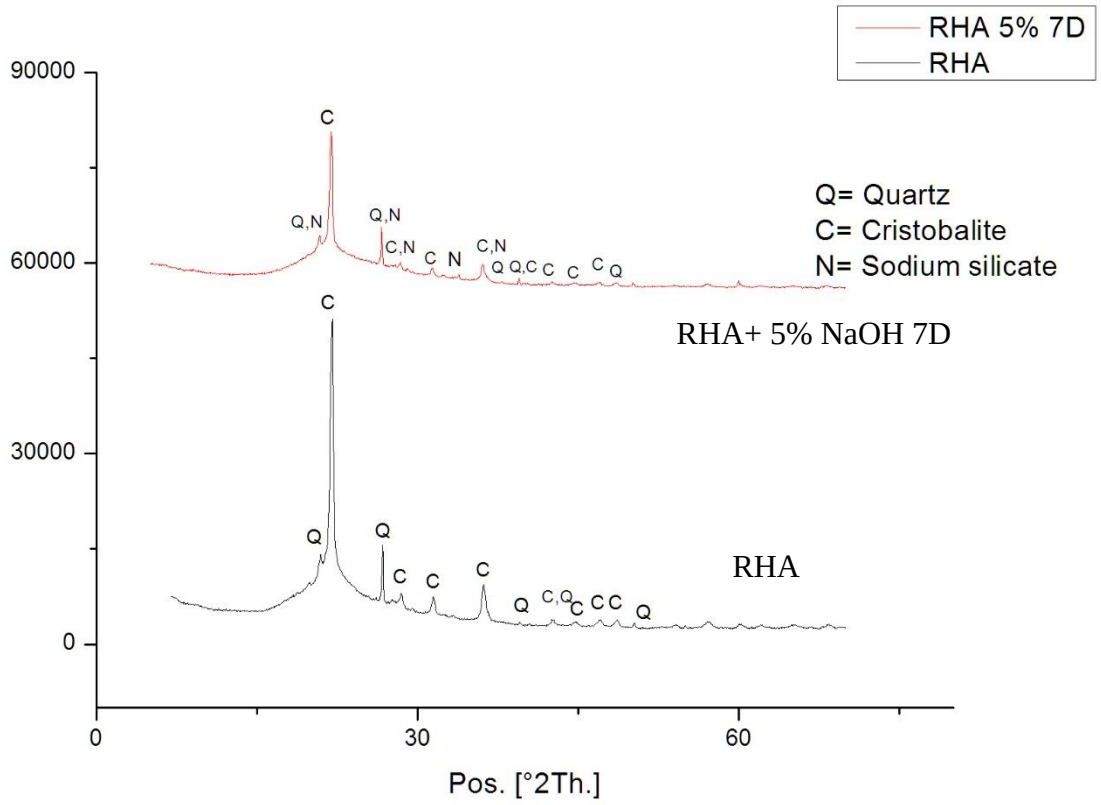


Figure 31: XRD pattern of RHA and activated RHA(5% NaOH) at 7 days

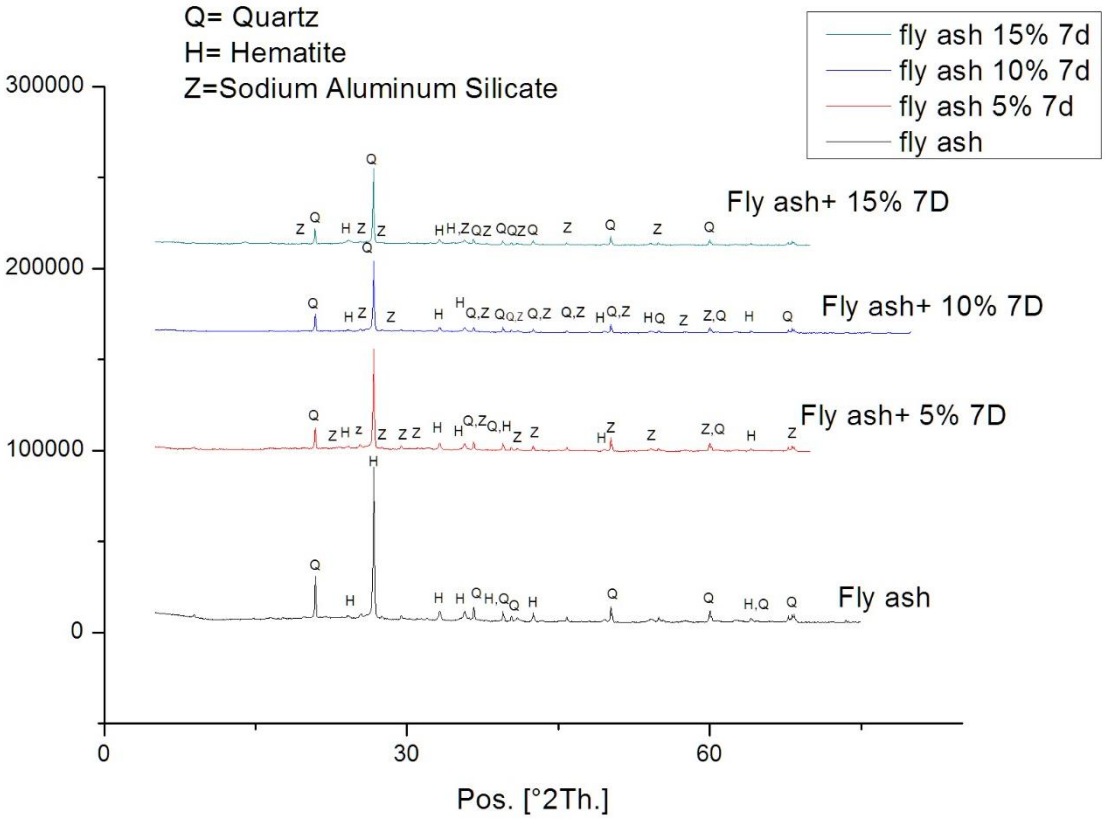


Figure 32: XRD pattern of fly ash and activated fly ash at 7 days

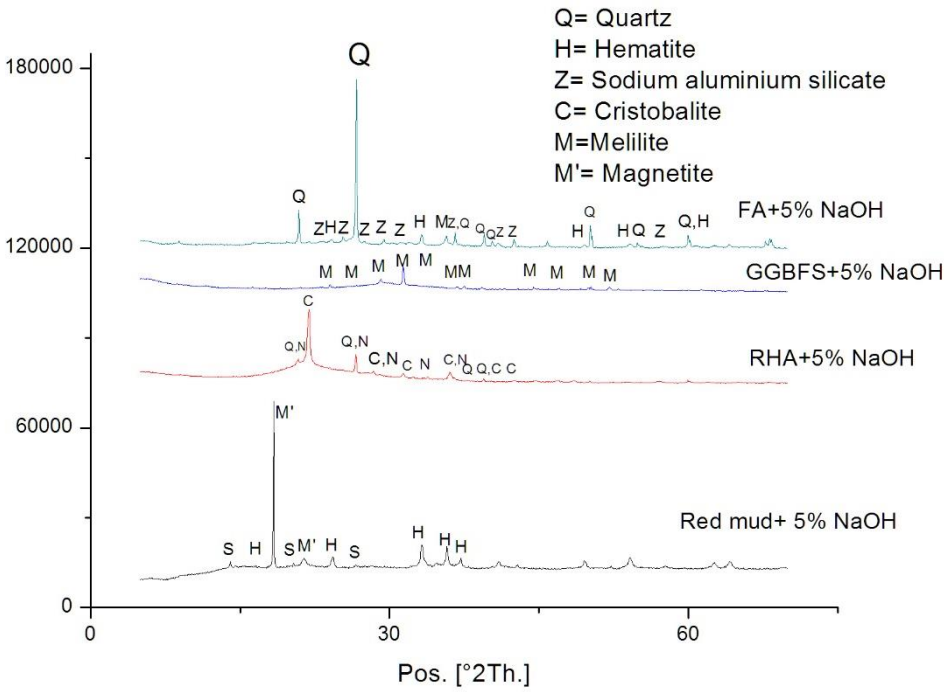


Figure 33: Comparison of XRD analysis of activated materials

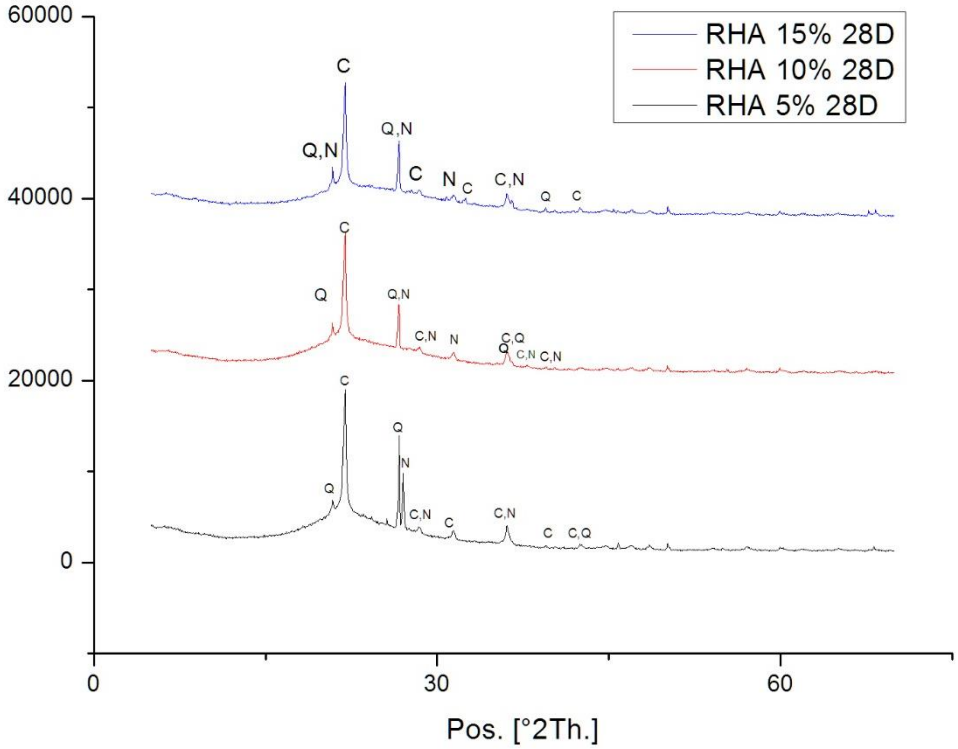


Figure 34: XRD pattern of activated RHA at 28 days

Chapter 5
Conclusion

5.1 Summary

The light compaction test, unconfined strength test and XRD analysis of the four waste materials (i.e. slag, fly ash, rice husk ash and red mud) were conducted in the project work. After that the raw materials were stabilized by using NaOH with different percentage. To know the effect of varying percentages of NaOH on by-products, unconfined strength test of the prepared samples were conducted on prefixed curing periods. To investigate the reaction product obtained after pre-decided curing periods, XRD analysis was done for some selected samples. Following conclusion are drawn from the experiments.

5.2 Conclusion

1. With increase in percentages of NaOH, the materials get more compacted due to the lubricating property of sodium hydroxide gel. Maximum dry densities of the materials increase with increase in NaOH % while optimum moisture content decreases. For slag, standard proctor test is not feasible for 15% NaOH and the sodium hydroxide solution becomes supersaturated and can't be mixed thoroughly.

2. The immediate strength of activated slag increase 2.89 and 7.6 times of the raw slag for 5% and 10% NaOH respectively. With increase in curing periods, the strength increases and for 28 days curing with 10% NaOH maximum strength is achieved around 27.5 MPa. It can be concluded from results that reaction between slag and NaOH occurs within 7 days of curing. There is no significant change in strength for 7 days and 28 days. Brittle failure of samples occurs on addition of NaOH.

3. In case of fly ash, the strength of samples with 5% NaOH is .55 MPa, which is 2 times more than the strength of raw samples. With increase in curing period, the strength of activated samples increase approximately 20 times the non-activated samples. At 28 days curing, for 10% and 15% NaOH samples, the gain in strength is almost same.

4. Decrease in immediate strength occurs when NaOH is added to Rice husk rice. Strength at 7 days curing is less than the immediate strength due to ongoing reaction between rice husk ash and NaOH. With increase in percentages of NaOH, the specimen bulged more during failure. With increase in curing time, the strength at 28 days increases from 7 days. Presence of carbon i.e. 10% prohibits the reaction between RHA and NaOH.

5. In case of red mud, increased strength occurs only for 5% NaOH additive at 7 days curing. On the addition of NaOH, the strength declines with curing periods. Decrease in results due to the presence of crystalline phases of material which forbids the reaction between.

6. Sodium hydroxide reacts rapidly with GGBFS than other materials (fly ash, RHA and red mud). The reaction between RHA particles and NaOH is too slow.

7. Formation of sodium aluminium silicate is observed in fly ash whereas sodium silicate gets developed in rice husk ash.

5.3 Scope of future work:

A detailed analysis of microstructure of alkali activated products is essential to understand the effect on the strength of by-products. Effect of temperature can be further investigated.

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